ABSTRACT

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In the presence of a catalytic system, an alcohol having "n" carbon atom(s) or a derivative thereof is allowed to react with carbon monoxide in a reactor 3 continuously, a higher bp catalyst component is separated from the resultant reaction mixture by a catalyst-separating column 5 to give a crude mixture, the crude mixture is fed to a higher bp component-separation column 8 to separate an overhead fraction from a bottom fraction containing at least a carboxylic acid having "n+2" carbon atoms, the overhead fraction is fed to a carboxylic acid-separating column 11, and are distilled in the presence of at least water and an ester of the carboxylic acid with the alcohol to separate a overhead fraction containing at least the ester and water from a bottom fraction containing the carboxylic acid having "n+1" carbon atoms. The overhead fraction from the carboxylic acid-separating column is fed to an aldehyde-separating column 14 to remove an overhead fraction containing an aldehyde. Such a process insures that impurities are efficiently separated from a reaction mixture by carbonylation of an alcohol, and that a carboxylic acid is purified easily at a lower cost.